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Reference Data for Orienting Quartz Plates by X-ray Diffraction

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The relative intensities of X-rays reflected from a large number of planes in quartz were measured. The results of these experiments are given with suggestions for their most effective use in the determination of orientation of crystal blanks. Graphs are presented which indicate the effect of inclined planes on the intensities of the reflected beam as well as the zero position of the Bragg angle reading. Tables and charts to facilitate the use of the most essential X-ray data are included. Other information of use to the technician in checking the orientation of crystal cuts is presented.

I. Introduction

Quartz crystals are used in communications and other electronic systems as frequency-controlling devices, filters, resonators, and electromechanical transducers. Because of the anisotropic structure of quartz it is possible by cutting at various orientations to obtain crystals suitable for these different applications. Each use is dependent to a large extent upon the orientation of the crystal cut with respect to the crystallographic axes. In order to achieve the desired effect the orientation must be determined with great accuracy. This is accomplished through the use of X-rays, which permit rapid and accurate checking of orientation by means of diffraction from the planes of the crystal lattice in accordance with the Bragg equation $n\lambda = 2d \sin \theta$, where n is the order of the reflection, λ the wavelength of the X-rays, d the interatomic plane spacing, and θ the angle these planes make with the diffracted X-rays. The d spacings are sufficiently different to permit identification of most planes by means of Bragg angle measurements, except when they are members of a "pair", for example, planes that have the hki^1 indices and l indices equal in magnitude but opposite in sign. For members of a "pair", the interplanar spacing is the same, but, fortunately, the reflection intensities differ sufficiently to permit positive identification. It is the purpose of this Circular to present the results of measurement of the relative intensities of X-ray beams reflected from a large number of planes in quartz, as well as other data and graphs that will be useful in establishing the orientation of quartz cuts.

2. Experimental Procedure

2.1. Preparation of Blanks

Brazilian quartz of high purity was used for the measurements. The blanks required were obtained from four different crystals. Careful checking of crystallographically identical planes

from a number of crystals, including the four used in the experiment, disclosed no apparent difference in reflection intensities. Bars whose angular tolerances were within 10' of specified directions were first made. Rough blanks were cut from the bars with their major surfaces approximately parallel to the desired crystal plane directions. The blanks were cut approximately $\frac{1}{4}$ in. thick to give them the necessary rigidity. They were ground by hand on No. 220 silicon carbide abrasive to remove all saw marks, eliminate other surface irregularities, and at the same time adjust their orientation to within $\pm 2'$ of arc of the desired orientation. They were then etched to disclose the presence of any twinned portions. A twin-free area approximately 1 in. square was cut from the rough blank. The trued surface was ground on No. 400 silicon carbide to remove all traces of etching, and the orientation rechecked to make certain it was within the tolerances specified above. A considerable period of time elapsed during the preparation of the blanks. In order to remove differences that might result from aging, all surfaces were freshly ground immediately prior to making the intensity measurements.

2.2. Measurement of Relative Intensities

The relative intensities of the reflected beams were measured on the General Electric XRD-1 X-ray unit equipped with a GE Crystal Goniometer. Radiation from a copper target, filtered through nickel, wavelength approximately 1.54 Å, was used. The reflected beam was detected and amplified by an ionization chamber and amplifier system (fig. 1). As the amplifier was nonlinear, it was calibrated in terms of known input voltages in order to secure linear relationship between the reflection intensities. These corrected values of intensity are given in table 1 and figure 2.

First-order intensities were measured for all planes except the 00·1, for which the third order was measured because the first and second are lacking. Second-order and first-order intensities were secured for 01·0, 01·1, and 01· $\bar{1}$ planes because second-order Bragg angles for these planes

¹ For an explanation of these symbols, refer to chapter II, Piezoelectricity, by W. G. Cady (McGraw Hill Book Co., Inc., New York, N. Y., 1946) and chapter III, section 5, Quartz crystals for electrical circuits, by R. A. Heising (D. Van Nostrand Co., Inc., New York, N. Y., 1946).

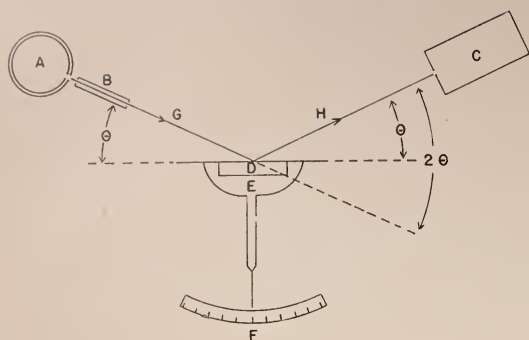


FIGURE 1. Diagram of X-ray goniometry.

A, X-ray tube; B, collimator tube containing two slit inserts and nickel filter; C, ionization chamber; D, quartz crystal; E, rotating crystal table; F, angular scale; G, incident ray; H, reflected ray.

often can be more conveniently used for orientation purposes. First-order reflections from these planes were off the scale of the meter. Therefore, a smaller slit, which reduced the readings 50 percent, was substituted for the regular slit. This brought the readings from the 01·0 and 01·1 planes on scale. Because the reflection from 01·1 still produced an off-scale deflection, it was necessary to reduce the intensity of the X-ray beam by decreasing the anode current from 23 to 13 ma.

3. Discussion

Figure 2 shows the orientation with respect to the Z-axis of planes parallel to the X-axis and gives for these planes the indices, Bragg angles, and reflection intensities. The positions of several common oscillator cuts whose faces are parallel to the X-axis are also shown. Table 1 gives the orientation with respect to the Z-axis of planes parallel to either the X- or the Y-axis,² with their indices, Bragg angle, and relative intensity. As was previously stated, inasmuch as members of pairs of planes have equal Bragg angles, it is often necessary to use intensity data to differentiate them, as for example, the 02·3 and 02·3̄. The Bragg angle is 34°5', but the reflection intensities are 33 and 89, respectively.

The intensity values given were obtained from planes parallel to the surface of the plate as in figure 3, a, and do not apply to planes nonparallel to the surface except for the case where the plane intersects the surface in a line that is parallel to

the plane of incidence (plane described by the incident beam and the normal to the plate, as in fig. 3, b).

When the crystal plane intersects the plate surface in a line that is perpendicular to the plane of incidence, the intensity of the reflected beam becomes stronger as the angle between the incident beam and the surface decreases, figure 3, c, reaching a maximum of twice the normal intensity when the angle of incidence is 0° and the angle of reflection 2θ. Conversely, the intensity decreases as the angle of incidence increases, becoming zero when the angle of incidence is 2θ and the angle of reflection is 0°.

The intensity is also affected by the condition of the surface. A coarsely ground surface gives a slightly stronger reflection than one finely ground. A deep etch reduces the intensity more than 50 percent. The relative intensities given in table 1 and figure 2 apply only when surface conditions are identical.

TABLE 1. Reflection intensities from atomic planes in quartz

Angle between plane and Z-axis	Indices h·k·l	Bragg angle λ=1.54 Å	Relative values of intensity
90°	00·3	25°20'	20
0°	01·0	10°26'	130
	02·0	21°14'	75
8°57'	05·1	65°27'	6
	05·1̄		1
11°8'	04·1	47°35'	3
	04·1̄		23
14°42'	03·1	34°10'	73
	03·1̄		2
17°29'	05·2	71°42'	1
	05·2̄		51
21°29'	02·1	22°55'	21
	02·1̄		43
27°42'	03·2	37°51'	22
	03·2̄		43
30°34'	04·3	57°17'	15
	04·3̄		24
	01·1	13°20'	364
38°13'	01·1̄		226
	02·2	27°27'	50
	02·2̄		17
46°23'	03·4	51°58'	9
	03·4̄		4
49°45'	02·3	34°5'	33
	02·3̄		89
52°41'	03·5	63°41'	1
	03·5̄		9
57°35'	01·2	19°45'	23
	01·2̄		52
63°4'	02·5	53°6'	2
	02·5̄		<1
67°3'	01·3	27°41'	34
	01·3̄		1
72°23'	01·4	36°15'	14
	01·4̄		37
75°45'	01·5	47°21'	26
	01·5̄		12
78°3'	01·6	60°59'	<1
	01·6̄		9
0°	11·0	18°17'	89
	11̄·0		92
24°27'	11·1	20°9'	27
	11̄·1		32
42°16'	11·2	25°5'	99
	11̄·2		99
53°45'	11·3	32°3'	21
	11̄·3		20
61°11'	11·4	40°37'	30
	11̄·4		29
66°15'	11·5	51°10'	14
	11̄·5		13
69°52'	11·6	65°41'	7
	11̄·6		7

² Y is not an axis of symmetry, and therefore faces or planes rotated 180° about Y do not belong to the same symmetry form. This means that the two opposite parallel faces of a slab of quartz cut parallel to the Y-axis are not crystallographically identical. Belonging to different forms they have different properties, etch patterns, etc. It is a well-recognized fact that crystal planes making the same angle with the Z-axis but belonging to different forms, as for example, the major and minor rhombohedral faces, may have markedly different intensities of reflection. In the course of this investigation it was found that faces which do not belong to the same symmetry form even though they constitute the opposite faces of a single blank may also reflect with different intensities. For example, the 11·1 plane of a slab reflects with an intensity of 32, whereas the opposite 11̄·1 plane parallel to it reflects with an intensity of 27. Blanks cut at other orientations around the Y-axis gave similar results. The differences, however, were much smaller, and in some cases were so small they were difficult to detect. (See table 1 and fig. 2.)

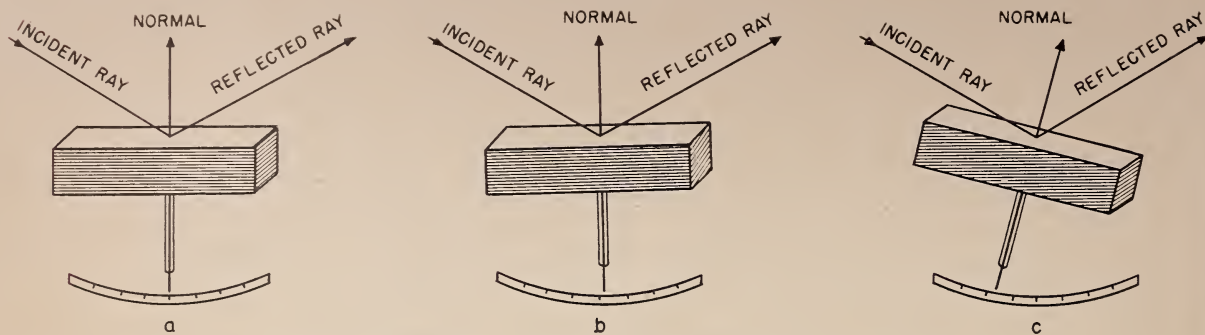


FIGURE 3. Positions of crystal planes relative to the surface of the blank.

4. Application

Table 2 gives the useful X-ray data concerning the most common quartz crystal cuts. The letters in columns 6 and 10 indicate the axis parallel to the plane of incidence of the beam during measurement of the reflection. The first three cuts listed are the basic *X*-, *Y*-, and *Z*-cuts whose major faces are normal to the axis used to designate them. For these cuts the crystal reference plane is parallel to the surface; therefore reflection occurs when the angle of incidence with the crystal surface equals the Bragg angle, as indicated in the sixth column. The remaining four cuts listed in column 1 are rotated *Y*-cuts having one edge parallel to the *X*-axis and the other edge rotated to a new direction *Z'*.

A possibility of error arises here since the major faces of these cuts are not parallel to the crystal reference plane. Two *Z'* readings are possible, equal to the Bragg angle plus or minus the angle between the plane and crystal face. Adding this angle when it should be subtracted and vice versa will result in cutting the crystal on the wrong side of the reference plane and cause an error in orien-

tation equal to twice the difference. Such errors may easily occur if the crystal face is within a degree of the reference plane. Checking from a second plane as indicated in column 10 will disclose whether this error has been made. If the blank is correctly oriented, the *Z'* readings obtained from the second plane will correspond to the reading having the same position in column 6. If incorrectly oriented, the *Z'* values given in column 10 will have an inverse relationship to those in column 6.

For example, the *BT*-cut crystal is inclined -49° to the *Z*-axis, whereas the 02.3 reference plane is inclined $-49^\circ 45'$. If the error previously mentioned occurs, the cut will be at $-50^\circ 30'$, and the resultant oscillator will not have the zero temperature-frequency coefficient normally associated with the *BT*-cut crystal. Checking the position of the test cut with reference to the 02.2 plane inclined at $-38^\circ 13'$ to *Z* will reveal the error. If the cut is accurately oriented, the angular difference will be $10^\circ 47'$, if not, $12^\circ 17'$ [$10^\circ 47' + (2 \times 45')$]. The correct *Z'* readings will occur at $38^\circ 14'$ and $16^\circ 40'$ and will be associated, re-

TABLE 2. Orientation data for common quartz cuts

Cut	Angle between cut and Z-axis	Angle between reference plane and Z-axis	Reference plane indices	Bragg angle	Actual reflection angle	Intensity	Angle between check plane and Z-axis	Check-plane indices	Actual reflection angle	Intensity
1	2	3	4	5	6	7	8	9	10	11
<i>X</i> -----	0°	0°	11.0	18°17'	$\left\{ \begin{array}{l} Y\ 18^\circ 17' \\ Z\ 18^\circ 17' \end{array} \right\}$	89				
<i>Y</i> -----	0°	0°	02.0	21°14'	$\left\{ \begin{array}{l} X\ 21^\circ 14' \\ Z\ 21^\circ 14' \end{array} \right\}$	75				
<i>Z</i> -----	90°	90°	00.3	25°20'	$\left\{ \begin{array}{l} X\ 25^\circ 20' \\ Y\ 25^\circ 20' \end{array} \right\}$	20				
<i>AT</i> -----	35°15'	38°13'	02.2	27°27'	$\left\{ \begin{array}{l} Z'\ 30^\circ 25' (27^\circ 27' + 2^\circ 58') \\ Z'\ 24^\circ 29' (27^\circ 27' - 2^\circ 58') \\ X\ 34^\circ 5' \\ Z'\ 33^\circ 20' (34^\circ 5' - 45') \\ Z'\ 34^\circ 50' (34^\circ 5' + 45') \\ X\ 27^\circ 27' \end{array} \right\}$	17	49°45'	02.3	$\left\{ \begin{array}{l} Z'\ 48^\circ 35' (34^\circ 5' + 14^\circ 30') \\ Z'\ 19^\circ 35' (34^\circ 5' - 14^\circ 30') \end{array} \right\}$	89
<i>BT</i> -----	-49°	-49°45'	02.3	34°5'	$\left\{ \begin{array}{l} Z'\ 33^\circ 20' (34^\circ 5' - 45') \\ Z'\ 34^\circ 50' (34^\circ 5' + 45') \\ X\ 27^\circ 27' \end{array} \right\}$	33	-38°13'	02.2	$\left\{ \begin{array}{l} Z'\ 38^\circ 14' (27^\circ 27' + 10^\circ 47') \\ Z'\ 16^\circ 40' (27^\circ 27' - 10^\circ 47') \end{array} \right\}$	59
<i>CT</i> -----	38°	38°13'	02.2	27°27'	$\left\{ \begin{array}{l} Z'\ 27^\circ 40' (27^\circ 27' + 13') \\ Z'\ 27^\circ 14' (27^\circ 27' - 13') \\ X\ 34^\circ 9' \\ Z'\ 37^\circ 20' (34^\circ 5' + 3^\circ 15') \\ Z'\ 30^\circ 50' (34^\circ 5' - 3^\circ 15') \end{array} \right\}$	17	49°45'	02.3	$\left\{ \begin{array}{l} Z'\ 45^\circ 50' (34^\circ 5' + 11^\circ 45') \\ Z'\ 22^\circ 20' (34^\circ 5' - 11^\circ 45') \end{array} \right\}$	89
<i>DT</i> -----	-53°	-49°45'	02.3	34°5'	$\left\{ \begin{array}{l} Z'\ 37^\circ 20' (34^\circ 5' + 3^\circ 15') \\ Z'\ 30^\circ 50' (34^\circ 5' - 3^\circ 15') \end{array} \right\}$	33	-38°13'	02.2	$\left\{ \begin{array}{l} Z'\ 42^\circ 14' (27^\circ 27' + 14^\circ 47') \\ Z'\ 12^\circ 40' (27^\circ 27' - 14^\circ 47') \end{array} \right\}$	59

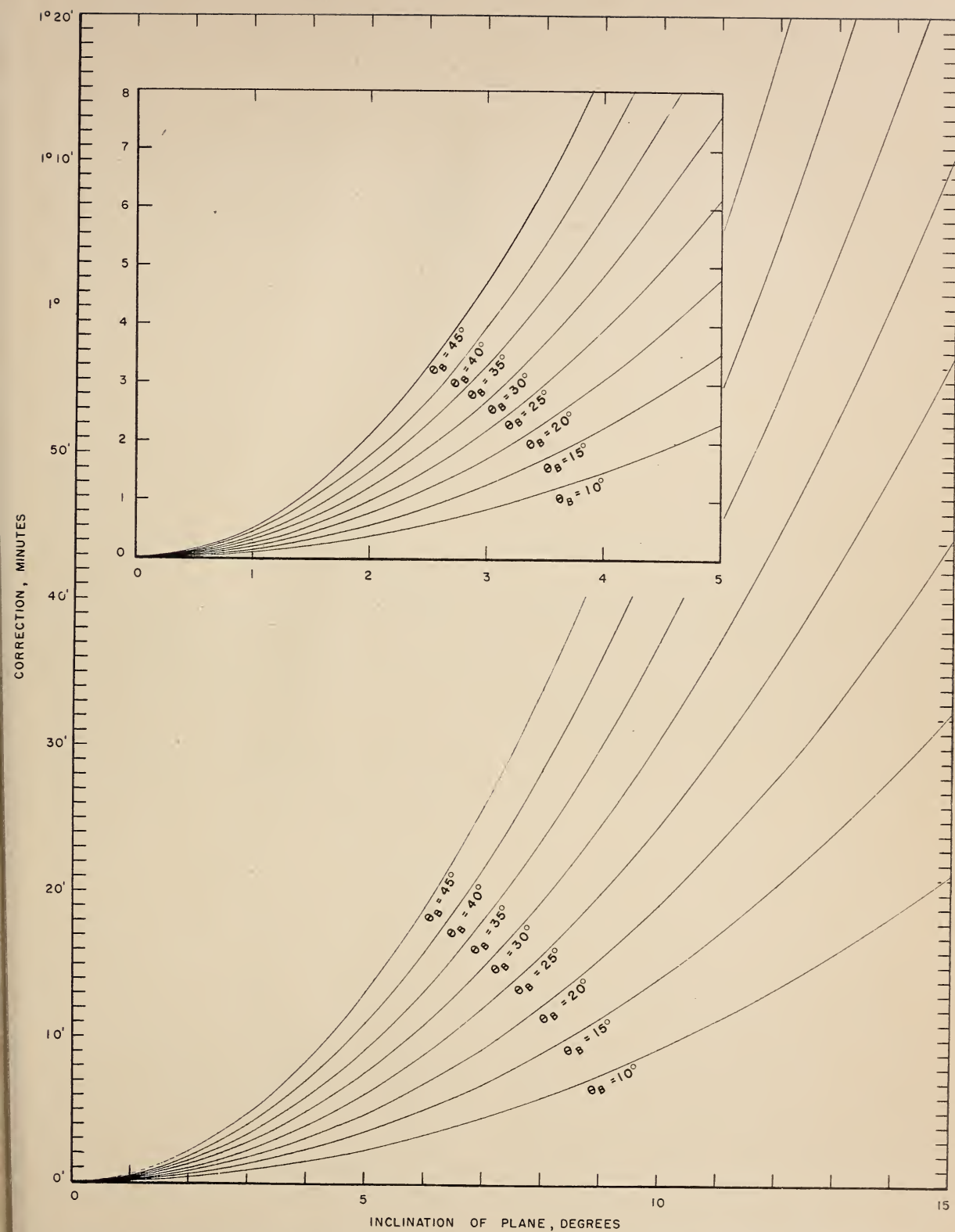


FIGURE 4. Graph showing the amount to be added to the angular reading when the reflecting plane is inclined.

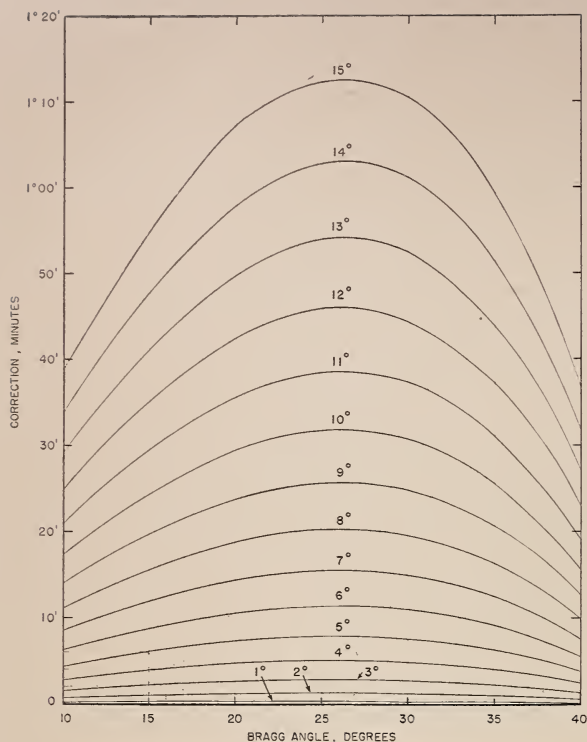


FIGURE 5. Graph showing the amount to be subtracted from the normal 2θ position of the ionization chamber with inclination of the reflecting plane up to 15° .

spectively, with the $33^\circ 20'$ and the $34^\circ 50'$ reflection from the 02.3 plane as given in column 6. If incorrectly oriented, the reading associated with $33^\circ 20'$ will be $15^\circ 10'$, and with $34^\circ 50'$ will be $39^\circ 44'$, which are not given in the table.

Another serious error may occur if the wrong member of a pair is chosen as the reference plane. As the d spacings are the same, reflection occurs at the same angle θ , but the orientation of the cut will be in error by twice the ZZ' angle. It is in cases like this that intensity values become significant because the large difference in intensity of the beams reflected from such planes usually provides an easy means for detection of the error should the wrong plane be selected. For example, as previously mentioned, the intensity from the 02.3 reference plane used for orienting the BT -cut is 33 while that from the 02. $\bar{3}$ is 89.

The intensity values given in column 7 for the planes listed in column 6 are the true values measured from planes cut parallel to the surface. The actual Z' readings will be found to deviate from these true values due to the effect described in section 3, paragraph 3. Table 2 gives data concerning the most common cuts in use. Supplementary data concerning reflection intensities from various other useful planes are given in table 1.

An error in orientation may arise from failure to realize that the forward or backward tilt of a plane affects the Bragg angle reading, even though

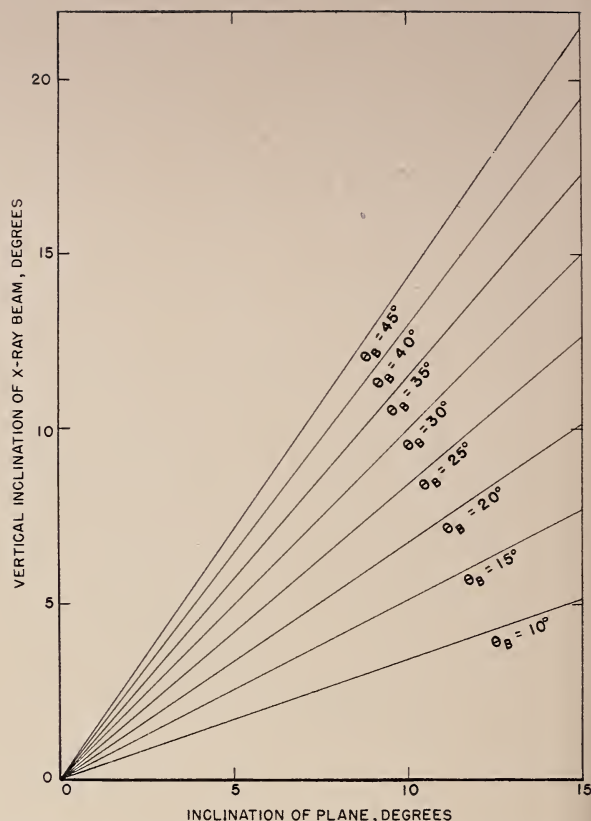


FIGURE 6. Vertical inclination of X-ray beam with inclination of reflecting plane.

the plane lies parallel to the upper and lower edges of the crystal blank, as shown in figure 3, b. In such cases the incident beam makes a larger angle with the surface of the crystal than it does with the plane. In order to obtain a reflection, the position of the crystal must be changed by an amount equal to the difference. This difference increases as the inclination of the plane from the perpendicular increases and is greater for large than for small angles θ . Figure 4³ indicates the correction to be made. In every case, it must be added to the Bragg angle. For example, a DT -cut crystal inclined at -53° to the axis oriented with reference to the 02.3 plane at $-49^\circ 45'$. When the blank is placed in the position illustrated in figure 3, b, the plane is inclined $3^\circ 15'$. From the graph it can be seen that the correction to be made is $4'$, indicating that the reading will be $34^\circ 9'$ instead of $34^\circ 5'$, the Bragg angle for that plane.

The inclination of the plane also affects the direction of the reflected beam. Not only is it deflected vertically by an inclined plane, but its

³ Calculated from formulas given in the chapter on X-ray techniques written by W. L. Bond and E. J. Armstrong, p. 95-139, "Quartz crystals for electrical circuits", by R. A. Heising (D. Van Nostrand & Co., Inc., New York, N. Y., 1946).

horizontal component does not make an angle 2θ with the incident beam, as shown in figure 1. It may therefore be necessary to adjust the position of the ionization chamber. The graph in figure 5 (see footnote 3) indicates the horizontal adjustment that must be made for various Bragg-angle and tilted-plane combinations. The adjustment necessary increases rapidly as the tilt of the plane increases, reaching a maximum for any inclination when the Bragg angle is approximately 26° . Figure 6 (see footnote 3) shows the vertical

inclination of the beam and is useful in indicating the degree of plane inclination that can be allowed with various Bragg angles. Maximum allowable vertical deviation is dependent on the length of the slit in the ionization chamber and may vary from 5° to 15° for different instruments.

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